

# OXYGEN DIFLUORIDE HANDLING MANUAL

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ALLIED CHEMICAL CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
CONTRACT NAS 3-2564 and NAS 3-6298
Theodore Male, Project Manager

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### FINAL REPORT

### OXYGEN DIFLUORIDE HANDLING MANUAL

by

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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NASA Lewis Research Center
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### **FOREWORD**

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### **ABSTRACT**

General information covering chemical and physical properties as well as appearance, solubility, and stability of OF2 are reviewed. Compatible and non-compatible materials, and proper cleaning and passivation techniques are discussed.

Health and fire hazards,  $OF_2$  toxicity and physiological effects as well as first aid are included together with methods for reducing hazards. Safety measures include protective clothing in addition to facility and personal safety equipment.

Decontamination methods to be used to neutralize spills and leaks, and the disposal of surplus OF2 are discussed.

The transfer and storage section includes materials, equipment, and procedures to be used for OF<sub>2</sub>. The applicable laws, marking and packaging procedures are also listed. Adequate references are provided throughout the report.

### 1. INTRODUCTION

Oxygen difluoride is a highly energetic, space storable oxidizer which possesses great merit as a rocket propellant. To utilize its great potential it is first important to acquire the knowledge which will permit it to be used effectively and safely. It is therefore essential that a manual be available as a reference for all phases of OF<sub>2</sub> handling.

This oxygen difluoride handling manual incorporates all pertinent available information so that it can serve as a convenient guide for OF2 handling procedures. This report is based on a review of the literature, discussions with persons experienced in this field, and also draws upon Allied Chemical Corporation's own vast experience as a manufacturer and supplier of this material. The similarity between OF2 and fluorine has led to a review of acceptable practices for fluorine handling. Pertinent information from this source has been included.

### 2. GENERAL PROPERTIES

Oxygen difluoride, OF2, is a powerful oxidizing agent which has been given serious consideration as a component of high-energy rocket propellant systems. It is a colorless gas at ambient conditions which condenses to a pale yellow liquid at  $-145.3^{\circ}$ C. It reacts readily with a majority of inorganic and organic compounds with high heats of reaction. Such reactions are often sufficiently energetic to cause ignition. Oxygen difluoride is hypergolic with many fuels and should be treated with appropriate precautions. Some physical properties of OF2 are shown in Table 1.

Oxygen difluoride is the simplest and most stable binary compound of oxygen and fluorine. Its behavior is quite similar to that of fluorine and the halogen fluorides. Although oxygen difluoride is considerably more toxic than fluorine, chemically it is less reactive. Compatibility studies have found OF2 to be less corrosive than fluorine. Therefore materials, equipment and handling procedures that are satisfactory for fluorine are generally equally applicable for oxygen difluoride.

TABLE 1
PROPERTIES OF OF2

Physical Properties		Reference
Molecular Formula	OF <sub>2</sub>	
Molecular Weight	54.00	
Freezing Point	-223.8°C (-371°F)	1
Boiling Point	-145.3°C (-230°F)	2
Critical Temperature	-58.0°C (-72°F)	3
Critical Pressure	48.9 atm. (719 psia)	3
Critical Density	0.533 g/cc	
Vapor Pressure	$\log p_{mm} = 7.2242 - \frac{555.42}{T^{\circ} K}$	2
	Range: -195°C to -145°C	
Density of Liquid	1.521 g/cc at its normal boiling point	3
	Density =2.190-0.00523 T° R	3
	Range: $-145$ to $-153$ °C	
Density of Gas	$0.0021$ g/cc at $21^{\circ}$ C	4
	$0.0023$ g/cc at $0^{\circ}$ C	
Viscosity, liquid	0.2826 centipoises at -145	5.3°C 3
	$\log n = \frac{131.5}{T^{\circ}K} - 1.5768$	
	Range: -146° to -153°C	
Heat of Vaporization	$2.650 \text{ kcal/mol at } -144.8^{\circ}$	5
Trouton's Constant	20.65	5
Solubility in Water	$6.8$ cc gas per $100$ cc of wat $0^{\circ}$ C, $11$ atm.	vater 5
Heat of Formation, gas	$\Delta \text{ H}_{\text{f298}}^{\circ} = +5.86 \pm .17 \text{ kca} 1/\pi$	nole 6

### 2.1 Chemical Properties

Oxygen difluoride is almost as powerful an oxidizer as fluorine but generally requires a higher activation energy to initiate its reactions. Reactions between OF2 and oxidizable materials often have high heats of reaction and many such reactions reach ignition. oxygen difluoride is quite stable at ambient temperature and can be stored in proper containers for long periods without noticeable decomposition. In the presence of water, OF2 will hydrolyze slowly forming oxygen and HF. OF2 is unstable at elevated temperatures, the decomposition rate is appreciable at approximately 250°C. The OF<sub>2</sub> dissociates into its constituent elements, oxygen and fluorine. Ice has been found to be shock sensitive in liquid OF2 and moisture therefore must be excluded from OF2 systems.

Oxygen difluoride is not shock sensitive. Sensitivity tests using Trauzl block techniques indicated OF<sub>2</sub> to be insensitive to shock at -196°C (Ref. 7). A cylinder of liquid OF<sub>2</sub> was subjected to gunfire which ruptured the cylinder but failed to detonate the OF<sub>2</sub> (Ref. 8). Investigations conducted under this contract indicated that gaseous OF<sub>2</sub> did not detonate or damage its container when it was instantly decomposed by thermal shock (Ref. 14).

### 3. <u>MATERIALS</u>

### 3.1 Metals

Many common metals and alloys have been found to be compatible with oxygen difluoride. The most widely acceptable materials are Monel, nickel, stainless steels (300 series), copper, and aluminum. These materials have proven to be satisfactory for both liquid and gaseous oxygen difluoride service. The actual choice of material should be governed by design requirements such as temperature, pressure, duration of exposure and flow velocity. Considerations should also be given to weight requirements and economic feasibility before selecting a material for a given situation.

Table 2 lists all metals which have been used or tested for  $0F_2$  service. For simplicity, the materials have been divided into three broad categories: compatible, not suitable, and those with insufficient background data to make a firm classification.

It should be noted that several materials have been placed into two categories. For example, aluminum alloys and nickel are so listed for dynamic service in liquid OF<sub>2</sub>. They are compatible inasmuch as they will not ignite or have a significant corrosion rate under these conditions. However, under very high

flow velocities (300 ft./sec.), test orifices have shown some enlargement. The use of aluminum under dynamic conditions should therefore be limited to either low velocity applications, as in pipe lines, and to situations where a very slight dimensional change can be tolerated. These materials would therefore not be recommended as orifices, or valve stems and seats. On the other hand, materials that are rated as suitable under static conditions can also be used under mildly dynamic conditions.

It must be emphasized that the compatibility or suitability of any material with  $OF_2$  is contingent upon its being properly cleaned and passivated prior to  $OF_2$  exposure.

# 3.2 <u>Non-Metallic</u>

# 3.2.1 Plastics

The performance of plastic materials in oxygen difluoride service has been erratic. Plastic materials should therefore be used only for limited applications since these materials are readily affected by OF2 under dynamic conditions. Additionally, all of the chemically inert plastic materials which have been tested have been impact sensitive in liquid OF2.

TABLE 2

COMPATIBILITY OF METALS WITH OF2 (REF. 7 to 13)

Code: X = Compatible; NS = Not Suitable; - = Insufficient Information

<u>Material</u>	Liquid		Gas	
	<u>Static</u>	Dynamic (a)	<u>Static</u>	Dynamic (a)
Aluminum Alloys				
A1 1100	X	X,NS	X	-
A1 2014	X	X,NS	X	-
A1 2020	X	X,NS	X	-
A1 2024	X	X,NS	X	X
A1 2219	X	X,NS	X	-
A1 3003	X	X,NS	X	-
A1 5052	X	X,NS	X	-
A1 5154	X	X,NS	X	-
A1 5456	X	X,NS	X	-
A1 6061	X	X,NS	X	X
A1 7075	X	X,NS	X	-
A1 7079	X	X,NS	X	-
A1 7178	NS	NS	-	-
Stainless Steels				•
SS 301 (FH)	X	X	X	X
SS 304 (FH	X	X	X	X
SS 309 Cb	X	X	X	X
SS 310	X	X	X	X
SS 316 (A & FH)	X	X	X	X
SS 347 (A & FH)	X	X	X	X
SS 410	X(1)	•	X(1)	-
PH-15-7 Mo	X	<u>-</u>	X	X
Maraging Steels		•		
AM 350	X	-	X	-
AM 355	X	-	X	-
AM 367	X	•	X	<del>-</del>

TABLE 2 (continued)

Material	Liquid		Gas (a)	
	Static	Dynamic (	a) <sub>Static</sub>	Dynamic (a)
Nickel Alloys				
Nickel 200,201,& 211	X	X,NS	X	X
Monel 400	X	X	X	X
Mone1 K-500	X	X	X	X
Monel, Cast	X	X	X	X
Inconel X	X	X	X	X
Inconel 600	X	X	X	X
Rene 41	X	-	X	X
Hastelloy D	X	-	X	-
Copper Alloys				
Copper	Х	X	X	X
Cufenloy 10	X	-	X	-
Cufenloy 40	X	(2)	X	-
Brass (70 - 30)	Х	-	X	-
Phosphor Bronze	X	X	X	X
Beryllium Copper (2% Be)	X	-	X	-
Chromium Copper (1% Cr)	X	X	X	X
Magnesium Alloys (3)				
Mg H-24	X	-	X	-
Mg HM 21A-T8	X(2)	_	X	-
Mg A231B	x`	-	X	· <b>-</b>
Mg FS-1A	X	-	X	-
Mg HK-31A	X	-	X	-
Titanium Alloys (3)				
Ti A-110AT	X(2)	X(2)	X	X
Ti 6 A1-4V	X(2)	-(2)	X	-
Ti A-55	-	-	X	_
Ti B120-VCA	_	_	X	-
Ti 16U-2.5 A1	_	-	X	-
Kentanium	Х	-	X	-

## TABLE 2 (continued)

Material	Liquid		Gas	
		Dynamic(a)	Static	Dynamic(a)
<u>Miscellaneous</u>				
Tantalum	X(2)	<b>-</b> (2)	X	-
Tin	NS	NS	NS	NS
Lead	NS(2)	NS(2)	$\mathbf{X}$	NS
Mild Steel	(1)	(1)	X(1)	-
Columbium (1)	X	NS	X	-
Brazed Monel (b)	、 X	X	X	X
Silvered Soldered Monel (c	) X	X	X	X
Welded Monel (a)	X	X	X	X
Platinum	-	-	X	-

- (1) Not suitable for cryogenic service.
- (2) Impact sensitive in liquid OF2.
- (3) Not all the listed materials have been tested for impact sensitivity. Those that have been tested and found not to be sensitive are aluminum alloys, stainless steels, copper alloys, nickel alloys and columbium. In view of the exhibited sensitivity of those titanium and magnesium alloys that were tested, similar alloys are suspect.
- (a) In excess of 150 ft./sec.
- (b) Brazing Rod, Oxweld #25, Linde Corp.
- (c) Silver Solder, Rod #1801, Eutectic Welding Alloys Corp.
- (d) Monel Filler Metal 40, International Nickel.

The three classes of plastics that appear to be most resistant to  $OF_2$  are listed below in order of preference: (Ref. 14)

- (a) tetrafluoroethylene (Halon TFE, Teflon TFE)
- (b) chlorotrifluoroethylene (Plaskon 2200, Kel F)
- (c) fluorinated ethylene propylene (Teflon FEP)

Specimens of these materials have been tested in liquid OF2 under both static and dynamic conditions

and were not chemically affected. However, these materials require a relatively low initiating energy to start a reaction which could result in the ignition and destruction of hardware. The reaction with slight surface contamination could provide this necessary energy. The margin of safety when using plastics is unknown but obviously small. This fact alone would indicate that plastics should be avoided, especially under dynamic conditions. In actual field service there have been numerous failures of gaskets, valve seats and O-rings fabricated of TFE, the best of the plastics. In many instances the plastic has simply disappeared.

There are some applications, however, in which TFE has shown great merit. It is the only acceptable valve packing for OF<sub>2</sub> service. Extensive use as a pipe thread compound has shown TFE pipe tape to be satisfactory for this application. One layer of tape should be wrapped tightly around all but the first two male threads of the member which is to be connected. When TFE is used as a valve packing, care must be taken that there are no leaks across the packing. TFE cold flows and packing glands require constant attention to prevent leaks, especially if the valve is cycled frequently.

While several other plastics have appeared to be resistant to OF2 on the basis of short term static tests, the use of plastics other than the three recommended classes is to be avoided. For example, Kynar which appeared to be unaffected by liquid OF2 is so severely embrittled at cryogenic temperatures as to be worthless. Viton 7250, as another example, was relatively unaffected by liquid OF2, but showed a very significant attack in OF2 gas at room temperature As a further precaution, it should be (Ref.14). remembered that a plastic to be used in OF2 systems must also have resistance to fluorine since it will be in contact with this gas when the system is passivated. This fact alone precludes the use of many plastics which might otherwise have been given additional consideration.

### 3.2.2 <u>Miscellaneous</u>

There are a few other non-metallic materials that appear to be resistant to  $OF_2$ . Again, the particular properties of these materials restrict their use to limited applications.

- (a) Pyrex glass resistant to both liquid and gaseous OF2 up to 200°C.
- (b) Sintered alumina (A1203) This includes materials such as alundum and sapphire.
- (c) Fused Si02 Quartz.
- (d) Fused metal oxides and fluorides cermets.

- (e) Ceramics
- (f) Permatex #2 Pipe thread compound for gaseous service at ambient temperatures.
- (g) <u>Fused carbides</u> Norton's Norbide (B4C) and Titanium Carbide cermet.

Other non-plastics should not be used without testing. Graphite, for example, is shock sensitive in liquid  $OF_2$  and explodes violently.

### 3.3 Cleaning

### 3.3.1 Metals

It is extremely important that all materials to be used in  $0F_2$  service must be carefully and thoroughly cleaned, and free of surface contamination. Many system failures and ignitions have been caused by inadequate cleaning. The cleaning procedure which follows is generally applicable to all metal components: (additional cleaning information may be found in References 7, 15 and 16)

- (1) All components should be as completely disassembled as is practical. Materials that are non-metallic such as valve packings should be removed and cleaned separately.
- (2) Gross contaminants such as grease, oil, burrs, scale, weld slag, fluxes, dyes, and other foreign matter should be removed by the most appropriate cleaning technique. Acid soaks for removal of scale and oxides, alkaline washes or soaps for the removal

of oils and greases, and abrasive treatment for surface stains are suggested treatments. The choice of the cleaning ingredients should depend on its compatibility with the particular material. Cleaning should be continued until the metal attains a bright surface. The cleaned components must then be thoroughly rinsed with water to remove any trace of the contamination and cleaning agents.

- (3) All components should be cleaned by vapor degreasing or by sonic washing in a detergent solution. The parts should then be carefully rinsed several times with hot water, followed by rinses with distilled or ionized water.
- (4) The water can be removed by washing with a solvent such as Genesolv DI, a mixture of 65% Genesolv D and 35% isopropanol. Final rinsing in Genesolv D or similar solvents is followed by drying in a vacuum oven at approximately 150°C. If the size of the component precludes drying in a vacuum oven, drying with heated dry nitrogen may be substituted.
- (5) Unless the components are to be used immediately, the cleaned and dried parts are to be packaged in clean plastics such as Aclar or polyethylene. Be sure that the packaging materials are equally as clean as the components to avoid contamination. If aluminum foil is used as a wrapping be certain that it too is clean and oil free.

(6) It is highly advisable that assembled systems be cleaned even though cleaned components have been used. Particulate matter generated in the process of assembly may both contaminate the assembly and cause mechanical problems (Ref.10). The cleaning of the assembled equipment can be accomplished by flushing with a halocarbon solvent such as Genesolv D until the spent solvent indicates the system is free of particulate matter. flushing is then followed by dry nitrogen or helium purging to remove the solvent. The final step is the evacuation of the system (to at least 1 mm) and sealing it off. Should the vacuum remain, it indicates that the removal of solvents has been complete, and of course, that the system is leak tight. A rise in pressure is a contrary indication. If the system is such that the solvent cannot be completely removed by purging or evacuation, it is better to omit the flushing. In this event, even more care should be given to the cleaning of the components and the succeeding assembly steps to

# 3.3.2 Non-Metals

Plastic materials are not cleaned in the manner that is acceptable for metals. Plastics such as TFE are slightly porous and have a tendency to absorb and hold cleaning solvents. These retained solvents may later become

avoid contamination.

sources of ignition. Therefore, it is generally preferred to wash plastics in soap and water. If solvents such as acetone are used it is best to provide a final rinse or a sonic wash in a Genesolv D or similar high purity solvent.

Drying should be done in a vacuum oven to make certain that all solvents have been desorbed.

Nitric acid washing of specimens of TFE, CTFE, and FEP, followed by water washes and solvent rinse may also be used. The specimens are then vacuum oven dried for several hours. Specimens thus cleaned were used successfully in dynamic tests with liquid OF<sub>2</sub> at 500 psig (Ref. 10).

Plastics require careful handling to avoid surface scratches and contamination. A fingerprint on an otherwise clean plastic surface may initiate ignition.

### 3.4 Passivation

After a system has been thoroughly cleaned and dried it is passivated with fluorine. This produces an inert fluoride film which inhibits further attack. Passivation should be considered a necessary and final step in the cleaning procedure since it tends to destroy or inert any minute contamination that may have escaped the previous cleaning steps. Under no circumstances can passivation be used as a substitute for good cleaning.

Passivation should always be performed with fluorine even though the system will be used for OF2 service. Fluorine, being more energetic, will react readily with both contaminants and hardware whereas OF2 may Before introducing the fluorine the system must be clean and dry. If possible, the system should be evacuated to remove moisture. If evacuation is not possible, thorough purging with dry nitrogen to remove all traces of moisture and solvents is indicated. Fluorine gas is then slowly admitted to the system to either relieve the vacuum or displace the nitrogen until the fluorine is quite concentrated. At this point, the fluorine pressure is slowly increased in several increments until system pressure or a minimum of 50 psig is reached. The actual pressure and the holding time is somewhat dependent on the system design. An intricate system with dead ends should be given a longer passivation period to assure the diffusion of the fluorine to all units of the system. Thirty minutes is a minimum period for passivation. Longer periods and higher pressures are suggested if the system is to be used for severe service. The optimum procedure is to passivate at the working pressure of the assembly. After the system has been passivated, the fluorine is vented and purged with dry nitrogen. Positive pressure should be kept on the system until it is to be used to

prevent the inadvertent entrance of moisture.

Moisture can destroy the passive film and in fact may greatly increase corrosion by the formation of HF (Ref. 15). Remember that passivation of individual components is neither necessary or satisfactory. Passivation is practical only when performed on the completely assembled system.

### 4. HAZARDS

### 4.1 <u>Health</u>

### 4.1.1 Inhalation - Toxicity

Oxygen difluoride is extremely toxic and the inhalation of this gas is to be avoided at all times. The tentative threshold limit value of 0.05 ppm has been established by the National Academy of Science - National Research Council Committee on Toxicology. It should be noted that this concentration cannot be detected by smell. The lower odor threshold is approximately 0.1 ppm although 0.5 ppm can be detected readily. The odor of very dilute concentrations of 0F2 is not too unpleasant and is somewhat like fluorine. In greater concentration the odor becomes foul and disagreeable. It has been reported that accommodation to the odor occurs rather rapidly. This again emphasizes the importance of leaving the contaminated area when the odor is first detected.

The recommended emergency limits for OF<sub>2</sub> exposure are: (Ref. 17)

60 minutes - 0.1 ppm

30 minutes - 0.2 ppm

10 minutes - 0.5 ppm

Unfortunately the  ${\rm OF}_2$  concentration cannot be determined by odor and therefore these numbers are merely significant in that they emphasize the hazard of  ${\rm OF}_2$  inhalation.

Toxicity studies (Ref. 18) conducted with rats indicate that a 50% mortality can result from a 5 minute exposure to 17 ppm or 15 minutes at 8 ppm of 0F<sub>2</sub> in air. These exposures produced widespread lung damage. Respiratory distress and death occurred within several hours and up to two days after exposure. It should be noted the test animals displayed no evidence of irritation during the period of exposure. It is therefore imperative that all inhalations or exposures to 0F<sub>2</sub> should be reported and the victim should remain under competent medical supervision for at least 24 hours since the respiratory effects may often be delayed.

# 4.1.2 <u>Skin Contact-Burns</u>

Contact with either liquid  $0F_2$  or a jet of gaseous  $0F_2$  may produce serious burns which resemble thermal burns. However, since the reaction of  $0F_2$  with skin and and tissue produces hydrogen fluoride (HF), treatment

of burns must consider the corrosive effect of the HF.

Burns caused by lower concentrations of  $OF_2$  more closely resemble HF burns and are treated as such. It must be remembered that HF, like  $OF_2$ , is insidious; symptoms may not develop for several hours. However, such burns are extremely painful and heal slowly.

### 4.1.3 Treatment

### 4.1.3.1 Inhalation

Any person who has been exposed to  $OF_2$  fumes should leave the contaminated area immediately. There is no specific prophylactic treatment for  $OF_2$  exposure. If there is difficulty in breathing or any respiratory distress, oxygen should be administered. The victim should be taken to a medical facility where accurate diagnosis can be made of any damage. It is important that the patient should be observed for 24 hours since lung damage or other symptoms may not develop for many hours after exposure. The medical treatment is then taken as the symptoms indicate.

A study of eighteen OF<sub>2</sub> inhalation incidents involving perhaps 25 persons indicated that almost as many "symptoms" were reported. In addition to respiratory distress, which covers irritation and breathing difficulties, other reported symptoms were headache, sleepiness, coughing, weakness, sore throat, shortness of breath, dizziness and the lingering slight odor of

OF<sub>2</sub> for many hours. In one case, the chemist developed pneumonia within 24 hours after exposure. He was hospitalized for an extended period before he fully recovered. A review of the reported symptoms does not indicate any set pattern or correlation of symptoms with one exception. Only two people reported headaches and each of these also stated they could smell OF<sub>2</sub> faintly for several hours after exposure. Since these two incidents were well isolated from each other, it would appear that these may also be legitimate symptoms of OF<sub>2</sub> inhalation.

### 4.1.3.2 <u>Contact</u>

All areas of the body that have been contacted by OF<sub>2</sub> should be copiously washed with water to remove all of the contaminant. Flushing should be maintained for a minimum of 15 minutes. Contaminated clothing should be removed during this period. The treatment used for HF burns should be followed. This consists of the application of iced alcohol or iced aqueous quaternary ammonium compounds, such as Hyamine, used as soaks or compresses. These treatments have been especially effective for the treatment of second degree HF burns (Ref. 19).

The Hyamine solution should be prepared in advance as a standard precaution when working with OF2. The solution is formulated as follows: Dissolve 2 grams of Hyamine 1622 in a liter of Formula 46 alcohol or in a liter

of distilled water. The patient should receive competent medical assistance as soon as possible after he has been burned. Subsequent treatment should be administered or recommended by a physician. Early treatment may prevent serious consequences and alleviate the extreme pain associated with HF burns.

### 4.1.3.3 <u>Eyes</u>

Special attention is required for any actual or suspected OF2 contact with the eyes. Liquid OF2 or vapors may produce irreparable damage unless prompt attention is given. The eyes should be flushed copiously for 30 minutes with clean water. The pain of such injuries may cause the patient to close his It is imperative that the eyelids be held apart so that the eyes and adjacent tissues will be properly flushed. No medication should be applied to the eye. Hyamine soaks which are recommended for burns on other parts of the body must not be used on or about the eye. An ophthamologist should be contacted immediately and any subsequent treatment should be performed as he directs.

# 4.2 <u>Fire and Explosions</u>

Oxygen difluoride is an extremely powerful oxidizer. Therefore contact with combustible materials will cause fires and possibly explosions.  $OF_2$  is not shock sensitive and explosions therefore must be the result of the reaction of  $OF_2$  and some highly oxidizable materials.

In the event that a fire occurs, an attempt should be made to shut off the source of OF<sub>2</sub>. It is advisable to have remotely operated valves in the system so that this can be accomplished quickly and safely. If the spill or leak can be shut off, the fire can be safely controlled with conventional extinguishants. Water spray deluge or fog can be safely applied to OF<sub>2</sub> spills and fires. The resultant HF formation as well as the toxicity of the OF<sub>2</sub> must, however, be considered. Therefore, personnel engaged in fire fighting must be equipped with suitable protective clothing and adequate breathing apparatus.

To minimize fire hazards, the facilities should be free of combustible materials. Construction should be of fireproof materials and combustible materials must be avoided. Good housekeeping should be practiced. The immediate areas should be free of debris and should not be used to store combustible materials or supplies.

Facility construction should be aimed at containing or restricting the  $OF_2$  in the event of a leak or spill. To this purpose, barricades of concrete, cinder block, or metal should surround storage tanks and test facilities. Spray heads or fog nozzles within the barricades should be capable of remote activation. The area could then be safely deluged in the event of a spill with minimal danger to personnel.

### 5. SAFETY

### 5.1 Personnel Protection

All personnel working with OF2 should be required to wear safety glasses and neoprene gloves which provide minimal protection. When working on or about the facilities containing liquid or gaseous OF2 at elevated pressures, a standard face shield should be For repairing leaks or breaking into any system that contains or has contained OF2, a clean neoprene protective outfit consisting of boots, gloves, face shield, jacket and trousers is required. outfit, together with a supplied air breathing apparatus is required when entering an atmosphere which contains detectable concentrations of OF, fumes. clothing should be loose fitting and quick opening so that it can be shed rapidly in the event it becomes contaminated or ignited. Protective clothing must be clean and dry to minimize the possibility of its reacting with  $OF_2$ . No clothing can be considered completely satisfactory against a jet of liquid OF2. Safety clothing should be considered only as short term protection under emergency conditions. The philosophy of protective clothing has been well stated (Ref. 16): "The use of protective clothing should be limited to those conditions where it affords protection. Extravagant use of protective clothing may provide a false sense of security, while in fact being only a physical hindrance."

### 5.2 <u>Facility Protection</u>

The protection of personnel starts with the proper facility design. Facilities should use fireproof construction, and the elaborate use of barricades and similar devices to control and minimize the effects of any accidental releases of OF2. Enclosed areas should be provided with extensive exhaust facilities with sufficient capacity to prevent the buildup of toxic concentrations of OF2 in the event of a minor leak. Consideration should be given to the location of exhaust exits. Vents which cannot be decontaminated should be well elevated to permit diffusion and dilution of toxic fumes. The location of vent exits should also consider the affects of possible contamination of adjacent areas. Facilities should also be equipped with remotely controlled spray deluge systems so that resultant fires can be rapidly and safely controlled. It is very important that all facilities be provided with several alternate methods of exit so that the entrapment of personnel is avoided.

# 5.3 <u>Safety Equipment</u>

In addition to the convenient location of lockers containing protective clothing and breathing apparatus, safety showers and eye wash stations must be provided. Such facilities should be located near enough to the work areas to be conveniently accessible to personnel working in the area. However, they must also be

beyond the reach of contamination in the event a gross equipment malfunction occurs. Shower facilities should also consider climatic conditions. The shock of a frigid shower may put an additional Therefore strain on an already injured person. tempered showers should be installed whenever possible. First aid facilities should be conveniently placed. First aid kits should contain the necessary supplies for HF burn 'treatment in addition to their normal materials. Emergency breathing oxygen should be available for treatment of  ${\tt OF}_2$  inhalations. important that all personnel working with OF? be aware of its toxicity and other hazards and be well trained in emergency procedures and the fundamentals of first aid for OF2 exposure. Personnel should not work alone in OF2 facilities and should always obey all safety rules and regulations.

Facilities should be equipped with alarm systems such as bells, horns, or lights so that personnel may receive adequate notice of any emergency. Hazardous test areas should be properly posted to prevent the entry of unauthorized personnel. Fume detectors may be used to monitor remote locations to give warning of leaks and spills. Particularly hazardous operations may be monitored by closed circuit television.

### 6. <u>DECONTAMINATION AND DISPOSAL</u>

### 6.1 <u>Spills</u>

The most effective decontaminant for liquid  $OF_2$  spills is a dilute aqueous solution of ammonia. A 5% solution applied as a spray deluge is quite effective in neutralizing the  $OF_2$  vapors above the spill and does not react violently with the liquid  $OF_2$ . The  $OF_2$  is converted to a relatively innocuous NH4F. Although water deluge can be safely applied to a spill, it is relatively ineffective as a decontaminant (Ref. 14).

Shower heads or spray nozzles should be strategically placed so that the entire spill area can be deluged. The spray discharge deluge should be remotely operated manually or by automatic devices activated by  $0F_2$  spills or fires. The spray equipment should be regularly and routinely tested by actual discharge to determine its operational effectiveness.

# 6.2 <u>Controlled Disposal</u>

Excess OF<sub>2</sub> should be decomposed and converted to innocuous compounds before release to the atmosphere. Several techniques have been developed for this purpose. Large quantities of OF<sub>2</sub> gas can be vented into a charcoal burner.

The toxicity of oxygen difluoride precludes direct venting into the atmosphere whenever it can be avoided. There are several techniques whereby waste OF2 can be decomposed and converted to relatively innocuous compounds which can then be vented. The reaction between OF2 and charcoal converts most of the fluorine values to CF4 which is inert and non-toxic. A refractory lined drum provided with a water-cooled inlet makes a satisfactory charcoal burner (Ref. 14). A 500 cubic foot reactor has been used to decompose fluorine (Ref. 16). The unit could probably be used equally well for the decomposition of OF2.

Large quantities of OF<sub>2</sub> can also be decomposed by venting it into an air deficient flame fed with natural gas, methane or propane. Although HF is one of the resultant by-products, the use of an exhaust stack permits the hot gases to rise and rapidly disperse in the atmosphere. The danger of HF contamination is negligible compared to the danger of venting toxic OF<sub>2</sub>.

Oxygen difluoride can also be destroyed by burning it with ammonia. One hundred pounds of  $OF_2$  can be consumed per hour in this manner. The user of this novel disposal technique considered it to be very effective, efficient and economical. The method was found to be equally suitable for fluorine disposal. The combustion of  $OF_2$  or  $F_2$  with ammonia at high feed rates is reported to be quite noisy. This disposal technique therefore may not be suitable at all locations.

### 6.3 Equipment

Equipment that has been used in OF<sub>2</sub> service or exposed to OF<sub>2</sub> vapors will be coated with fluoride films. Such films react with moisture and form HF. To avoid HF burns such equipment should be handled with rubber gloves. Water washing will safely and completely decontaminate the equipment so that it can be safely handled.

### 7. TRANSFER AND STORAGE

### 7.1 <u>Materials of Construction</u>

Although a great many materials are compatible with OF2 (Ref. Section 3), from a practical standpoint, construction materials are generally limited to a few classes of alloys. Nickel alloys have shown the greatest resistance to attack by OF2. Monel is the best alloy in this class and is acceptable for all Stainless steels, however, are OF<sub>2</sub> applications. the most widely used alloys for  $OF_2$  service and have generally been found to be as satisfactory as Monel. Stainless steel alloys are chosen for OF2 service because they are more readily available and more economical than Monel. Aluminum alloys are used where service requirements are not severe. weight advantage of aluminum is, of course, a strong factor in its selection. Other materials that may be used for OF<sub>2</sub> facilities include copper, copper alloys, titanium and magnesium. Since titanium

and magnesium alloys do show slight impact sensitivity in liquid  $OF_2$ , their use in static applications such as storage tanks is not encouraged. Table 3 lists various components of an  $OF_2$  system together with satisfactory materials of construction for both cryogenic conditions (liquid and gas) and ambient conditions (gas only).

### 7.2 Equipment

### 7.2.1 <u>Valves - Liquid OF</u><sub>2</sub>

Annin valves have been widely used for liquid OF2 service and their performance has been excellent. Generally, 1" valves with stainless steel bodies and copper seats are used, although Monel bodies have been selected when the need for additional safety factors can justify the extra cost. Teflon seats have been investigated but their performance has been erratic. The seats have often burned or simply disappeared. Copper seats on the other hand have given excellent service under very high pressures and velocities. The model to be selected is, of course, dependent upon the service requirements. For all models, however, a bellows type seal is preferred. Bellows of Monel or stainless steel are satisfactory. By back pressurization of the bellows the working pressure of the valve can be extended. Bellow valves have been used successfully at 1500 psi by this expedient (Ref. 10).

	(Ref.11 & 16)	
TABLE 3	MATERIALS OF CONSTRUCTION	TO THE OTHER

	7	
<u>Usage</u> Storage tanks	Cryogenic Temperature Stainless steel (300 series) aluminum, Monel,titanium,magnesium	Ambient Temperature Stainless steel (300 series), Monel, aluminum, mild steel
Lines & fittings	Stainless steel, Monel; flared and compression fittings	Stainless steel, copper, Monel, aluminum, mild steel
Gaskets	Soft copper,aluminum or nickel	Halon TFE or Teflon TFE if recessed and out of line of flow, copper & aluminum preferred
Regulators,flow meters	Standard metal orifices; turbine meters	Standard pyrex rotameters; S.S., aluminum or sapphire floats
Pressure gauges	•	All welded metal Bourdon tubes; stainless steel diaphragm and transducer (Barksdale type)
Injectors	Stainless steel (300 series); copper; nickel	1
Valve bodies	Nickel, stainless steel (300 series) Monel, aluminum, Inconel, copper (low pressure)	Nickel, stainless steel (300 series), Monel, aluminum, Inconel, brass
Valve plugs	Stainless steel (300 series), Monel	Stainless steel (300 series), Monel, bra
Valve seats	Nickel, copper, aluminum, brass	Nickel,copper,aluminum,brass
Valve packing	Not recommended	TFE (Halon or Teflon)
Valve caskets	Soft aluminum or copper	Soft aluminum, soft copper, TFE, lead
Bellows	Monel and stainless steel 347	Monel and stainless steel 347
Diaphragms	Stainless steel 347	Stainless steel 347
Thread sealants	Brazing,welding,silver solder	Brazing,welding,silver solder, TFE pipe tape
Bolts, nuts & screws	Stainless steel (300 series), Inconel X, Monel	Stainless steel (300 series), Inconel X, Monel
Rearings	Aluminum 6061, hard anodized copper	Aluminum 6061, hard anodized copper
Springs	<pre>K-Monel, Inconel X, stainless steel (300 series)</pre>	K-Monel, Inconel X, stainless steel (300 series)
Electrical Insulation	A1203	$A120_3$ , Pyrex

Valves suitable for cryogenic service other than Annin can, of course, be used provided that they too follow certain principles:

- a) The valve must have no plastics or elastomers that can be contacted by  ${\tt OF}_2$ .
- b) Seals are effected by metal gaskets and bellows.
- c) Valve construction should be such that it can be disassembled and thoroughly cleaned.
- d) All valves must be capable of remote controlled operation.

Some other valves that have given satisfactory service in liquid  $0F_2$  are Futurecraft No. 30354, and Marotta Valve, Model MV510X.

## 7.2.2 <u>Valves - Gaseous OF</u><sub>2</sub>

For gas phase service many other valves, in addition to those suitable for cryogenic service, have been satisfactory. Gas phase service is herein construed as handling OF<sub>2</sub> at approximately ambient conditions. Typical valves used in such service are Hoke 343 and 344 which employ a TFE packing. Valve bodies of Monel or stainless steel are both satisfactory. It should be noted that these Hoke valves have the packing beneath the stem threads. Valves with threads below the packing are not suitable since the valves must be cleaned and free of lubricant before going into OF<sub>2</sub> service. The unlubricated threads gall and seize and the valves become unusable. Other valves recommended for OF<sub>2</sub> gas

service include Matheson type 940F, and Whittaker Valve No. 230105. Packless valves such as Nupro SS-BW, with a welded bellows, as well as Hoke 440 and 470 series bellows valves have also proven satisfactory. Comparable valves employing the principles cited above would also be acceptable provided that they too were completely cleaned and passivated before going into service. Lastly, it should not be overlooked that bronze Chlorine Institute valves have been standard equipment on  $OF_2$  cylinders since Allied Chemical Corporation first packaged this gas.

## 7.2.3 Connections, Lines and Fittings

Welded connections are to be preferred whenever practical but are especially important for liquid OF2 dynamic service. The necessity of welding increases directly with the severity of the operating conditions. Flange closures using copper or aluminum gaskets may be used where frequent and easy disassembly is required. gaskets up to 10" diameter have been used with complete satisfaction. However, gaskets have a tendency to leak when the system is cycled between ambient and cryogenic temperatures. Constant pressure checking is therefore required. Welded closures eliminate this problem. Heliarc or inert gas welding is preferred. Welding techniques should be used which will prevent the formation of oxides that may subsequently react vigorously when exposed to OF2.

Brazing, and to a lesser extent silver solder, are also suitable methods for effecting leak tight connections. Fluxes used in these processes, however, may produce hazardous residues which must be carefully removed.

Other closures and connections have been used successfully in liquid OF<sub>2</sub> service. Monel Swagelok fittings up to 1/2" have been satisfactory at 1500 psi. Copper Swagelok fittings have been used at 1500 psig for OF<sub>2</sub> gas service. Copper Flare fittings (45°) have been used to a lesser extent in gas handling service generally at 400 psi and less. Parker AN fittings have been satisfactory for gas phase service up to 1000 psi. Successful service from these various fittings depends upon exercising the proper care in assembly. When tubing must be flared it must be done properly. The tubing must be free of burrs and die marks. Dell seals over smooth flares provide improved performance.

Lines and fittings are generally fabricated from stainless steel (300 series), Monel, nickel, copper and aluminum. The choice of material is dictated by the design conditions. All five materials have been satisfactory for both liquid and gaseous OF2. It cannot be over-emphasized that satisfactory performance depends on proper cleaning and passivation prior to actual use.

### 7.2.4 Metering

The preferred devices for the metering of liquid OF<sub>2</sub> are 400 or 300 series Fischer-Porter turbine type flowmeters with stainless steel vanes and bearings. Potter flowmeters, type P7D or P3D with stainless steel vanes and sapphire bearings have also been satisfactory.

Pressure differential cells have been used in conjunction with orifices to measure flow. Oil filled transducers have been used as pressure pickups but the potential hazard from an oil leak argues against their use. For metering  $0F_2$  gas at ambient temperature a pyrex rotameter is quite satisfactory. Gross transfer of gas from cylinders can be accurately measured from cylinder pressure drop since  $0F_2$  closely follows ideal gas laws.

## 7.2.5 <u>Pressure Gauges</u>

Pressure gauges constructed with welded Monel, stainless steel or bronze Bourdon tubes have all given satisfactory service with  $OF_2$  gas. It is imperative that the Bourdon tubes are LOX clean and passivated with fluorine before being placed in  $OF_2$  service. A gauge with a Monel Bourdon tube has been used at 7000 psig with  $OF_2$ . Gauges with stainless steel tubes have been used at 2000 psig.

### 7.2.6 Cryogenic Storage Vessels

Storage vessels for liquid  $0F_2$  must, of course, be fabricated from materials that are serviceable at cryogenic temperatures. Monel, stainless steel (300 series), and aluminum are the best materials for this service. Although several methods can be used to maintain the  $0F_2$  as a liquid, the preferred vessel consists of three concentric shells. The innermost shell contains the  $0F_2$  and the middle shell contains a refrigerant such as liquid nitrogen (LN<sub>2</sub>). The outer shell, which can be mild steel, is filled with an insulating material and evacuated. By replenishing the LN<sub>2</sub> as required,  $0F_2$  can be maintained as a liquid indefinitely.

Well insulated containers equipped with an internal refrigeration coil or an external condenser can also be used for cryogenic  $OF_2$  storage.

# 7.3 <u>Procedures for Transfer</u>

## 7.3.1 Liquid OF<sub>2</sub>

The transfer of liquid OF<sub>2</sub> can be readily accomplished by pressurizing the storage tank with helium. Provided that the transfer lines are properly insulated and the proper cryogenic type valves are employed, transfer is then simply a matter of valve manipulation. As a precaution it is good practice to provide double valving for all operations involving the transfer of liquid OF<sub>2</sub>.

Thus, in the event one valve leaks or fails, a leak or catastrophic spill can be prevented. In transfer operations, it is necessary to evacuate the receiver before admitting  $OF_2$ . Air will condense in liquid nitrogen cooled receivers. Liquid air is completely miscible with  $OF_2$  and will therefore act as a diluent. To prevent this, only helium, which is neither condensable at  $LN_2$  temperature nor soluble in  $OF_2$ , should be used as a pressurizing agent for liquid  $OF_2$ .

# 7.3.2 Gaseous $OF_2$

The transfer of  $OF_2$  from cylinders or storage vessels is very simple. Cylinders can be manifolded in large numbers to supply large amounts of  $OF_2$ . Each cylinder contains  $OF_2$  at 400 psig. Therefore transfer can be readily accomplished as long as a pressure differential exists between cylinder and receiver. If the  $OF_2$  is being condensed care must be exercised to prevent forming a vacuum in the cylinder which could cause the suck-back of air or material from the system into the cylinder. Moist air in a cylinder or system would destroy the passivating film and cause corrosion, and must be avoided at all times. Cylinders should be double valved and the main cylinder valve fitted with a device which will permit remote operations.

#### 8. PACKAGING AND SHIPPING

Oxygen difluoride is packaged as a compressed gas in seamless steel cylinders. The largest cylinders in current use are 10 5/8" outside diameter and 55 1/2" long, with a tare weight of approximately 195 pounds. This cylinder has a capacity of 9 pounds of oxygen difluoride gas at 400 psig. A similar cylinder of slightly smaller diameter has a capacity of 6 3/4 pounds of  $OF_2$  at 400 psig. The tare weight of this smaller cylinder is approximately 145 pounds. Both cylinders have a single outlet, valved with a Chlorine Institute valve having a nominal 1.030" diameter left hand external threaded outlet. A suitable adapter, designated as F 70M, is available from the Matheson Co., Inc., East Rutherford, New Jersey. This unit, made of Monel, can be used with lead gaskets where it seats onto the valve outlet. Soft copper and soft aluminum gaskets are also suitable.

Oxygen difluoride is shipped in DOT 3AA 1800 steel cylinders as a "Flammable Gas-N.O.S." Cylinders bear the DOT red label as well as the manufacturer's (Allied Chemical Corporation) label which provides adequate pertinent precautionary information.

Interstate shipments of cylinders are permitted via truck, rail and ship. However, trucks are the usual form of transportation. Cylinders must be firmly secured during shipment to prevent falling or rolling.

There are no restrictions on shipment other than meeting the necessary requirements for transporting a red labeled cylinder.

Oxygen difluoride can also be packaged and transported as a liquid in refrigerated tanks if large quantities are required.

Additional information as to cost and availability should be obtained from the Industrial Chemicals
Division, Allied Chemical Corporation, the only large producer and supplier of oxygen difluoride.

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